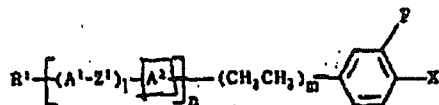


90-186489/25 E14 103 (E13) **MERCK PATENT GMBH**
 07.12.88-DE-841120 (+DE-939116) (13.06.90) C07c-255/50
 C07d-309/04 C07d-319 C07d-335/02 C07d-339 C09h-19/14
 2-Fluoro-benzonitrile deriva. prodn. - by metallation of 3-substd.
 fluoro-benzene(s), followed by carboxylation or formylation and
 conversion into nitrile
 C90-680922

(1) 2-Fluorobenzonitrile deriva. (I) of formula (I) (X = CN)
 are produced by metallation of the corresp. fluorobenzene
 deriv. (II) (X = H) to form the corresp. metal deriv. (III)
 (X = Li, Na or K), then carboxylating (III) and converting
 the carboxylic acid into (I) or formulating (III) and
 converting the resulting aldehyde into (I).



R¹ = up to 18C perfluoroalkyl, alkyl or alkenyl (opt. with
 one or more CH₂ gps. replaced by O, S or -C≡C-;

E(7-D5, 10-A15B, 10-A15D) L(3-D1D1)

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A¹, A² = (a) 1,4-phenylene, (b) 1,4-cyclohexylene (opt.
 with 1 or 2 non-adjacent CH₂ gps. replaced by O
 or S) or (c) 1,4-cyclohexenylene, 1,4-bicyclo-
 [2.2.2]octylene or piperidine-1,4-diyl (with (a)
 and (b) opt. mono- or poly-substd. with 10l
 and/or Me);

Z¹ = -CH₂-CH₂-, -OCH₂-, -CH₂O- or single bond;
 n = 0, 1 or 2; 1, m = 0 or 1; 1 + m = 1, 2, 3 or 4.

(3) Cpds. (I) with A¹ = A² = 1,4-phenylene, Z¹ =
 -CH₂-CH₂- or single bond, 1 = n = 1, m = 0 or 1 (if m = 0,
 then Z¹ = -CH₂-CH₂-) are claimed as such.

USE/ADVANTAGES

(I) are useful as intermediates, esp. for the synthesis
 of liq. crystalline cpds., or as components of liq. crystalline
 phases (to improve dielectric anisotropy or other properties).
 The invention provides a regiospecific process for the
 prodn. of (I) in good yield.

DETAIL

(II) is, e.g. 4-R¹-3'-fluorobiphenyl, 3-fluoro-4'-R¹-
 cyclohexylbenzene, etc. (16 R¹-substd. starting cpds.

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listed); metallation is carried out at -80 to -30°C with, e.g.
 n-BuLi, KNH₂, etc. in a solvent such as THF, dioxan,
 cyclohexane, toluene, etc. with HMPT etc. as activator.

EXAMPLE

10 ml. 0.1-M soln. of n-BuLi in hexane was added at
 -70°C to a mixt. of 0.1 mol. 3-(trans-4-heptylcyclohexyl)-
 fluorobenzene (prepd. as described in EP-116756) and 0.1
 mol. tetramethylethylenediamine in 200 ml. THF, then the
 mixt. was stirred for 2 hrs. at -70°C, treated with a mixt.
 of 0.1 mol. N-formylpiperidine and 20 ml. THF, warmed to
 room temp. and worked up to give a solid prod.; 0.1 mol.
 of the aldehyde obtd. was reacted with 0.12 mol. hydroxyl-
 amine O-sulphonic acid as described in Helv. Chim. Acta 59,
 2786 (1976) and worked up to give 4-(trans-4-heptylcyclo-
 hexyl)-2-fluorobenzonitrile as a colourless solid. The prod.
 had crystalline/nematic transition pt., 19°C; nematic/
 isotropic transition pt., 28°C. (16pp1712DWdWgNo4/0).

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